Ethephon

Collaborative Study

Full scale collaborative trial on the method for the determination of Ethephon in TC, TK and SL

5316/R

Report to CIPAC
By
Chinese Pesticide Analytical Committee（CHIPAC）

Method Developed by Shaoxing Eastlake High-Tech Co., Ltd

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Full scale collaborative trial on the methods for the determination of ethephon in TC, TK and SL

1. Ethephon description

ISO common name

No ISO common name. The common name of ethephon was approved by American National Standards Institute (ANSI).

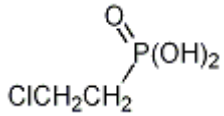
Synonyms

none

Chemical name(s)

IUPAC (R)-2-[4-(6-chlorobenzoxazol-2-yloxy)phenoxy]propionic acid ethyl ester CA ethyl (R)-2-[4-[(6-chloro-2-benzoxazolyl)oxy]phenoxy]propanoate

Structural formula



Molecular formula

C2H6ClO3P

Relative molecular mass

144.5

CAS Registry number

16672-87-0

CIPAC number

373

2. Ethephon method description

2.1 Outline of method

The content of ethephon is determined by ion chromatography using sodium carbonate and sodium hydrogen carbonate as eluent.

2.2 Apparatus and reagents

High performance ion chromatograph equipped with an electrolytic conductivity detector and an injection system capable of injecting 25 μl.

Electronic integrator or data system

Ion Exchange column: Dionex IonPac AS23, 250 x 4.0 mm (i.d.), or equivalent

Guard column: Dionex IonPac AG23, 50 x 4.0 mm (i.d.), or equivalent.

Analytical balance, accurate to ± 0.1 mg

Sodium carbonate: AR or GR

Sodium hydrogen carbonate: AR or GR

Ultrapure water

2.3 IC condition

Eluent:	7.2 mMNa_2CO_3+9.0 mM $NaHCO_3$
Flow rate:	1.0 mL/min
Current of inhibitor:	70 mA
Temperature of detector cell:	35℃
Temperature of column:	30℃
Mode of injection:	PushFull
Volume of Injection:	25μL
Frequency of data sampling:	5.0 Hz
Run time:	13 min
Retention time:	9.5 min

2.4 Procedure

(i) Preparation of Calibration solution. Prepare calibration solutions in duplicate. Weigh (to the nearest 0.1 mg) sufficient reference standard (w mg) to contain about 120 mg (ideally between 112 and 130 mg) of Ethephon into a volumetric flask (100 ml). Add ultrapure water to the mark and mix thoroughly. Transfer 5.00 mL of the above solution into a 50 mL volumetric flask, add ultrapure water to the mark and mix thoroughly. (Calibration solutions CA and CB).

(ii) Preparation of Ethephon sample. Prepare sample solutions in duplicate for each sample. Weigh (to the nearest 0.1 mg) sufficient sample (w mg) to contain about 120 mg (ideally between 112 and 130 mg) of Ethephon into a volumetric flask (100 ml) (for TC, melt the sample at 95℃ until the sample becomes a transparent liquid and mix well). Add ultrapure water to the mark and mix thoroughly. Transfer 5.00 mL of the above solution into a 50 mL volumetric flask, add ultrapure water to the mark and mix thoroughly. (Sample

solutions S1 and S2). Filter through 0.2 µm filter before use.

(iii) Determination of ethephon

(a) Equilibration of the system. Pump sufficient mobile phase through the column to equilibrate the system. Inject 25 µl portion of calibration solution CA until the response obtained from two consecutive injections deviate by less than 1.5%. Then inject 25 µl portion of calibration solution CB. The response factor for this solution should not deviate by more than 1.5% from that for calibration solution CA, otherwise prepare new calibration solutions.

(b) Determination. Inject in duplicate 25 µl portions of each sample solution bracketing them by injections of the calibration solutions as follows: calibration solution CA, sample solution S1, sample solution S1, calibration solution CB, sample solution S2, sample solution S2, calibration solution CA, and so on. Measure the relevant peak areas.

(iv) Calculation

Determine the peak area of Ethephon and calculate the mean value of response factors from the calibration solutions bracketing the injections of the sample solutions and use this value for calculating the Ethephon content of the bracketed sample solutions. The Ethephon content is the mean value of two sample solutions.

$$f_i = \frac{s \times P}{H_s}$$

$$\text{Ethephon content} = \frac{f \times H_w}{w} \quad \text{g/kg}$$

where:

f_i = individual response factor
f = mean response factor of bracketing calibration injections
H_s = peak area of Ethephon in the calibration solution
H_w = peak area of Ethephon in the sample solution
s = mass of Ethephon reference standard in the calibration solution (mg)
w = mass of sample taken (mg)
P = purity of Ethephon reference standard (g/kg)

3. Participants and sample distribution

Participants

Name of Laboratory	Country	Address	Contact
Bayer AG, Product Chemistry Analytics 1	Germany	Building 6510, 2.08 40789 Monheim, Germany	Dr. André Althoff
Laboratory for Quality Control of Pesticides National Phytosanitary Authority	Romania	11 Voluntari Bvld. VOLUNTARI Romania	Ciotea Florentina
FMC Corporation	USA	Stine Research Center 1090 Elkton Road Bldg. 315 Room 2224 Newark, DE 19711 USA	Mary Ellen P. McNally, Ph.D.
Shenyang SYRICI Testing Co., Ltd. China	China	No.8, Shenliao East Road, Tiexi District Shenyang 110021, P.R. China	Haixia Wang
Institute for the Control of Agrochemicals, Ministry of Agricultrue and Rural Affairs	China	Maizidian street 22, Chaoyang District, Beijing, P. R. China	Kaiwei Shi
Institute of Agro-product Safety and Nutrition, Zhejiang Academy of Agricultural Sciences	China	1-5040, New Area of Zhejiang Academy of Agricultural Sciences, No. 198, Shiqiao Road, Hangzhou, Zhejiang, China	Jianzhong Yu
Hunan Research Institute of Chemical Industry Testing Technology Co., Ltd.	China	No. 550, Changsha Avenue, Lituo street, Yuhua District, Changsha City, Hunan Province, China	Lu Huang
Guizhou Jiandee Technology Co., LTD	China	Baijin road No.3491,Baiyun district, Guiyang, P. R. China	Zhiyu He
Guizhou Testing Technology Research and Application Center	China	No. 388, Baisha Road, Baiyun District, Guiyang City, Guizhou Province, China	Rui Wang
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Laprode (Zhejiang) analysis Co., Ltd , China	China	4/F, Building 6, No.503 Xingguo Road, Yuhang District, Hangzhou, Zhejiang P.R. China	Aiping Xu

Jiangsu Agrochem Laboratory Co., Ltd, China	China	No.98, Minjiang Road, Hi-Tech Development Zone Changzhou, Jiangsu, China	Wendy Wang
Shaoxing Eastlake High-Tech Co., Ltd	China	No.359, Jiangzhong Road,Doumen Street, Yuecheng District, Shaoxing, Zhejiang	Xiaoying Ji
BioGuide Technologies Co., Ltd.	China	Buiding 8, IFST-CAAS, 2 Yuanmingyuan West Road, Haidian District, Beijing 100193, China	Chengjian Xia

Sample information

Sample	Quantity	Batch	Declared Content of AI
ethephon TC1	50 g	E202105089	Min. 93%
ethephon TC2	50 g	E202105091	Min. 93%
ethephon TK1	50 mL	F202105089	75%
ethephon TK2	50 mL	F202105091	75%
ethephon SL1	50 mL	202105089	40%
ethephon SL2	50 mL	202105091	40%

4. Deviations and remarks

Deviations:

Lab 4 set the temperatures of detector cell and column to room temperature, instead of 35℃ and 30℃.

Lab 8 set the temperature of detector cell to 30℃, instead of 35℃.

Lab10 used AS18 column, which is not AS23, and the flow rate was 0.34 mL/min.

Lab 12 used a flow rate was 0.7 mL/min, and the temperatures of detector cell and column were 40℃ and 45℃, instead of 35℃ and 30℃.

Lab14 used AS18 column, which is not AS23, and the eluent was 23.0 mM KOH, instead of 7.2mM Na_2CO_3 + 9.0 mM $NaHCO_3$.

Because the deviations of Lab 14 were significant, and its results contained multi-outliers, it was excluded from further statistical analysis.

Comments:

Two laboratories commented on the sampling method of TC: The samples could solidify

quickly if taken out of water bath for weighing, so it had better been kept in the water bath during weighing. An improvement to the sampling method may be considered.

5. Statistical evaluation

Table 1. Results of the analysis of Al content in the TC1

Lab	Day1 (g/kg)		Day2 (g/kg)		Average Yi(g/kg)	Yi^2	Standard Deviation Si	Si^2
	1	2	1	2				
Lab1	922.1	921.0	928.9	930.0	925.5	856515.0	4.5962	21.1246
Lab2	923.7	931.4	930.4	930.9	929.1	863216.3	3.6219	13.1183
Lab3	943.1	944.6	944.4	949.0	945.3	893566.7	2.5707	6.6083
Lab4	948.1	941.5	936.5	940.0	941.5	886507.9	4.8596	23.6161
Lab5	943.5	940.2	938.6	945.5	942.0	887273.5	3.1262	9.7733
Lab6	939.1	947.9	935.7	943.6	941.5	886484.9	5.3106	28.2028
Lab7	939.3	938.6	938.5	937.9	938.6	880935.3	0.5815	0.3381
Lab8	937.0	938.3	933.5	942.5	937.8	879524.5	3.7301	13.9139
Lab9	934.5	940.3	933.9	943.6	938.1	879980.9	4.6919	22.0138
Lab10	934.8	935.8	933.4	932.1	934.0	872412.8	1.6039	2.5724
Lab11	912.0	911.0	913.0	911.8	911.9	831593.7	0.8188	0.6704
Lab12	930.3	931.0	935.5	932.8	932.4	869395.3	2.3277	5.4183
Lab13	957.3	974.0	976.2	960.9	967.1	935254.8	9.4069	88.4891

Table 2. Results of the analysis of Al content in the TC2

Lab	Day1 (g/kg)		Day2 (g/kg)		Average Yi(g/kg)	Yi^2	Standard Deviation Si	Si^2
	1	2	1	2				
Lab1	923.4	919.3	930.2	941.9	928.7	862488.4	9.8668	97.3528
Lab2	929.1	929.3	927.7	915.7	925.5	856486.9	6.5625	43.0661
Lab3	943.7	941.0	944.8	940.7	942.5	888367.5	2.0158	4.0634
Lab4	940.5	950.4	945.5	950.5	946.7	896327.9	4.7629	22.6852
Lab5	948.3	938.5	939.6	946.4	943.2	889664.7	4.8678	23.6957
Lab6	939.9	932.5	939.6	935.7	936.9	877838.1	3.5134	12.3440
Lab7	938.8	938.6	937.9	938.0	938.3	880470.6	0.4539	0.2060
Lab8	938.0	939.5	937.8	935.0	937.6	879070.0	1.8719	3.5041
Lab9	940.8	930.1	932.5	926.4	932.4	869453.1	6.1008	37.2200
Lab10	934.2	935.4	931.1	932.4	933.3	871026.9	1.9011	3.6142
Lab11	909.8	914.3	914.3	910.4	912.2	832120.9	2.4795	6.1481

Lab12	938.6	940.7	934.6	935.8	937.4	878787.1	2.7496	7.5602
Lab13	881.3	933.1	940.6	933.1	922.0	850116.1	27.4019	750.8634

Table 3. Results of the analysis of Al content in the TK1

Lab	Day1 (g/kg)		Day2 (g/kg)		Average Yi(g/kg)	Yi^2	Standard Deviation Si	Si^2
	1	2	1	2				
Lab1	762.9	767.1	763.3	749.6	760.7	578716.0	7.6344	58.2845
Lab2	751.2	750.8	751.1	752.9	751.5	564791.7	0.9580	0.9178
Lab3	760.7	759.5	760.6	756.3	759.3	576484.6	2.0756	4.3083
Lab4	758.0	755.5	754.7	749.9	754.5	569321.2	3.4127	11.6466
Lab5	749.0	735.9	745.7	749.4	745.0	555041.8	6.2910	39.5768
Lab6	770.6	767.9	762.9	765.3	766.7	587770.7	3.3241	11.0499
Lab7	756.1	754.8	754.1	755.5	755.1	570216.2	0.8751	0.7659
Lab8	760.7	757.5	765.7	761.9	761.5	579808.1	3.3671	11.3376
Lab9	757.1	763.2	757.4	765.4	760.8	578789.6	4.1793	17.4666
Lab10	772.9	776.0	772.6	771.0	773.1	597684.9	2.0647	4.2630
Lab11	752.1	749.7	745.8	742.7	747.6	558854.3	4.1286	17.0454
Lab12	762.2	760.8	755.5	751.1	757.4	573668.3	5.0893	25.9012
Lab13	779.9	788.5	775.4	779.9	780.9	609821.8	5.4491	29.6931

Table 4. Results of the analysis of Al content in the TK2

Lab	Day1 (g/kg)		Day2 (g/kg)		Average Yi(g/kg)	Yi^2	Standard Deviation Si	Si^2
	1	2	1	2				
Lab1	760.4	756.8	775.6	766.8	764.9	585071.8	8.2737	68.4540
Lab2	761.7	764.3	759.3	764.6	762.5	581339.5	2.4578	6.0406
Lab3	765.1	763.3	762.7	765.1	764.1	583790.4	1.2273	1.5063
Lab4	761.2	756.0	759.7	758.0	758.7	575660.2	2.2321	4.9822
Lab5	738.2	738.0	745.6	743.2	741.3	549455.7	3.7780	14.2732
Lab6	763.3	766.0	764.7	769.8	765.9	586664.6	2.7812	7.7353
Lab7	755.2	755.2	755.2	755.2	755.2	570321.3	0.0408	0.0017
Lab8	758.1	762.4	767.8	767.4	763.9	583572.7	4.6280	21.4184
Lab9	765.6	766.8	768.9	765.8	766.8	587959.9	1.5293	2.3389
Lab10	770.2	772.9	773.6	772.8	772.4	596546.2	1.5090	2.2771
Lab11	747.5	751.3	743.9	745.0	746.9	557865.4	3.2666	10.6704
Lab12	769.7	768.1	756.5	756.6	762.7	581721.6	7.1687	51.3909
Lab13	784.8	783.2	763.0	788.3	779.8	608133.8	11.4061	130.1002

Table 5. Results of the analysis of Al content in the SL1

Lab	Day1 (g/kg)		Day2 (g/kg)		Average Yi(g/kg)	Yi^2	Standard Deviation Si	Si^2
	1	2	1	2				
Lab1	411.9	415.1	405.3	408.1	410.1	168178.7	4.2792	18.3117
Lab2	398.1	408.7	401.4	401.7	402.5	161985.1	4.4700	19.9810
Lab3	420.7	422.1	418.0	411.3	418.0	174735.2	4.7889	22.9338
Lab4	417.3	414.6	407.9	411.6	412.9	170454.1	4.0150	16.1199
Lab5	391.9	398.9	388.2	400.1	394.8	155836.8	5.6673	32.1186
Lab6	408.3	409.3	410.2	411.7	409.9	168009.3	1.4224	2.0232
Lab7	408.3	408.3	408.0	409.5	408.5	166907.4	0.6722	0.4518
Lab8	406.4	404.9	408.4	410.4	407.5	166049.0	2.3903	5.7138
Lab9	406.4	404.3	407.6	411.6	407.5	166030.0	3.0819	9.4979
Lab10	413.1	412.2	415.7	415.0	414.0	171377.6	1.5967	2.5494
Lab11	402.1	402.6	400.1	399.7	401.1	160899.1	1.4593	2.1296
Lab12	403.2	404.5	405.2	406.7	404.9	163940.5	1.4552	2.1177
Lab13	419.4	423.4	415.5	413.0	417.8	174573.9	4.5521	20.7215

Table 6. Results of the analysis of Al content in the SL2

Lab	Day1 (g/kg)		Day2 (g/kg)		Average Yi(g/kg)	Yi^2	Standard Deviation Si	Si^2
	1	2	1	2				
Lab1	404.0	404.7	416.1	407.8	408.2	166594.7	5.5653	30.9730
Lab2	401.9	395.1	398.5	400.1	398.9	159110.3	2.8570	8.1622
Lab3	417.9	419.9	418.2	415.6	417.9	174646.7	1.7531	3.0734
Lab4	414.4	412.9	408.2	411.7	411.8	169567.0	2.6295	6.9140
Lab5	396.3	397.8	402.1	399.4	398.9	159112.9	2.4990	6.2451
Lab6	409.1	409.8	409.8	411.4	410.0	168113.9	0.9696	0.9402
Lab7	405.0	405.4	404.6	403.3	404.6	163690.8	0.9161	0.8393
Lab8	404.8	406.4	407.9	408.5	406.9	165577.3	1.6577	2.7480
Lab9	407.6	403.4	408.9	403.7	405.9	164753.1	2.7666	7.6540
Lab10	409.2	409.0	405.2	411.3	408.7	167014.7	2.5538	6.5220
Lab11	404.6	403.8	398.3	400.7	401.8	161474.8	2.9227	8.5420
Lab12	404.1	402.6	402.2	401.8	402.7	162145.9	0.9950	0.9900
Lab13	418.2	422.3	410.8	413.5	416.2	173223.8	5.0740	25.7459

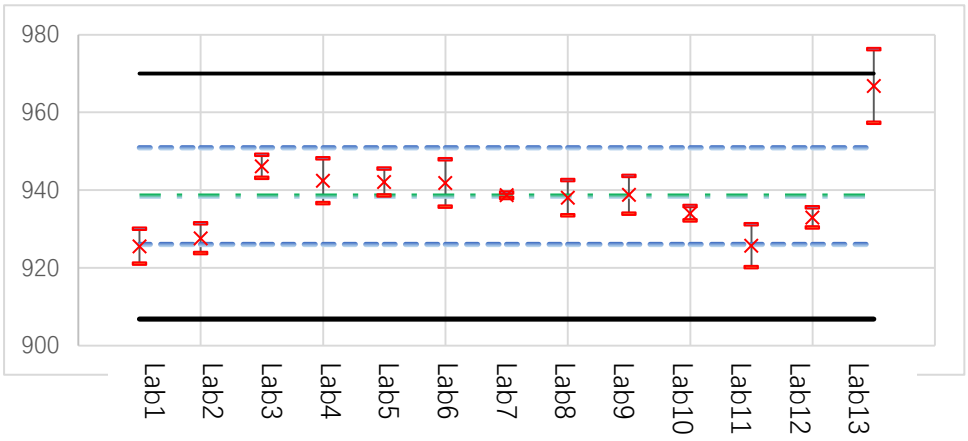

Figure 1. Graphical presentation of TC1 data

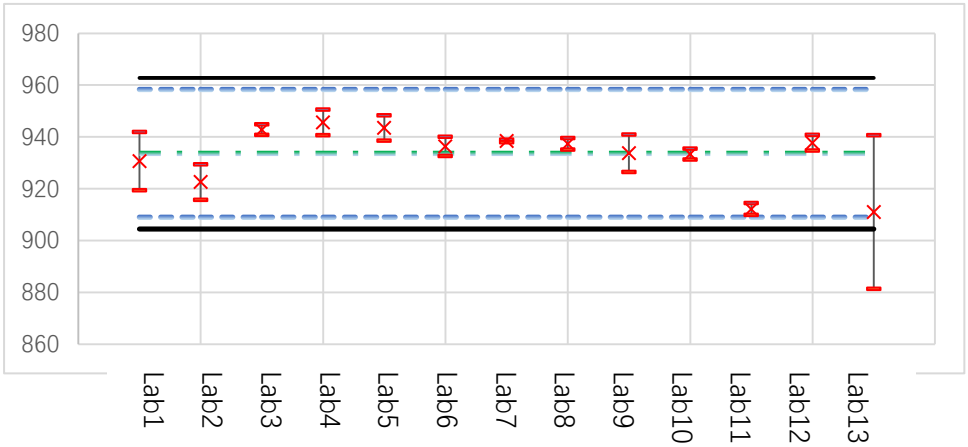

Figure 2. Graphical presentation of TC2 data

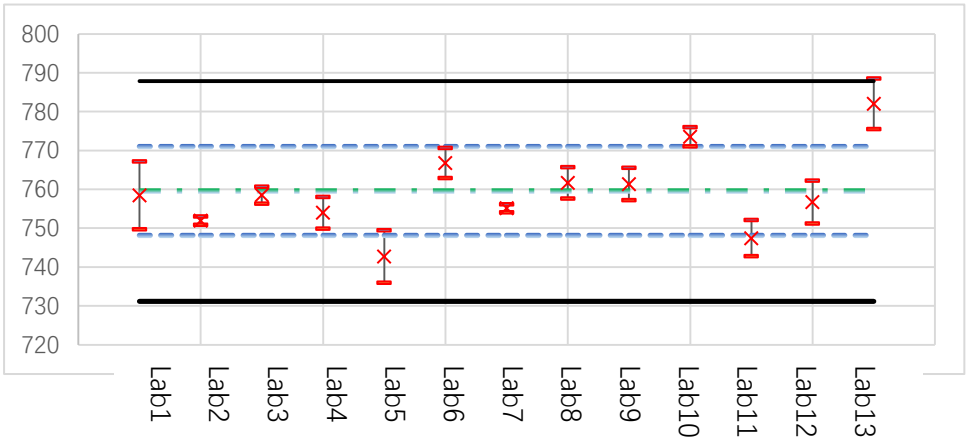

Figure 3. Graphical presentation of TK1 data

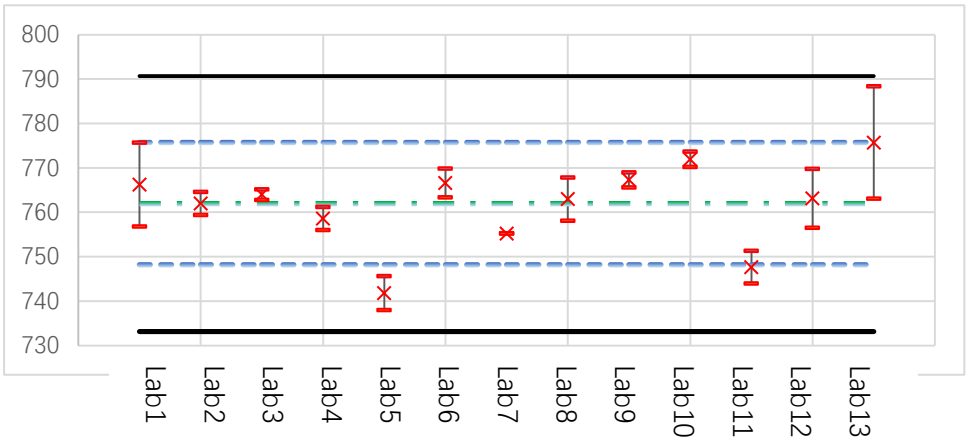

Figure 4. Graphical presentation of TK2 data

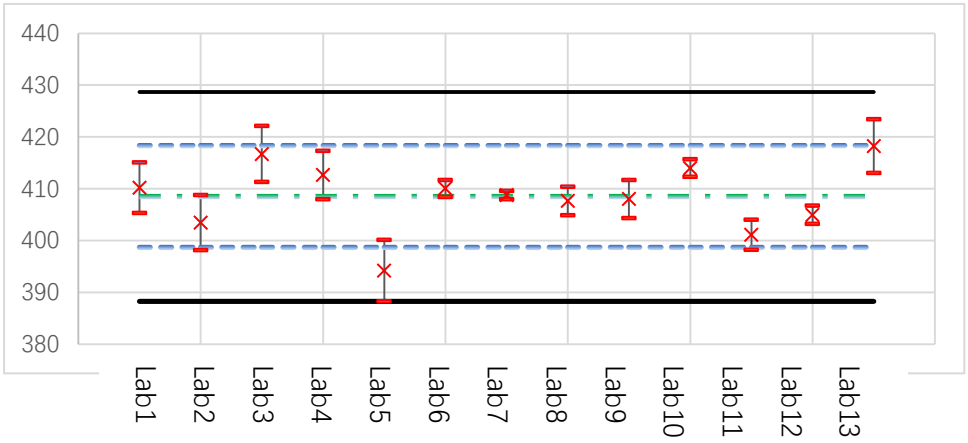

Figure 5. Graphical presentation of SL1 data

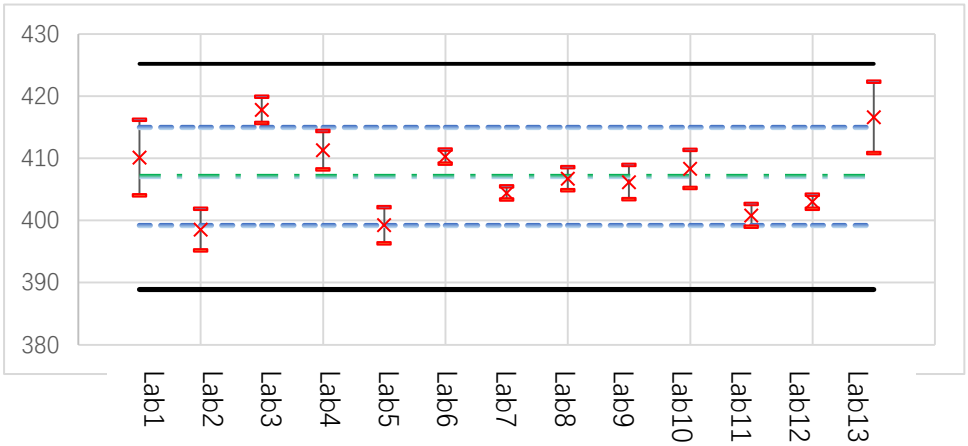

Figure 6. Graphical presentation of SL2 data

Table 7. Statistics of the results of TC1

S_1=sum Yi	12199.41		
S_2=sum Yi^2	11449470.13278		
S_3=sum Si^2	256.5940		
No. Lab P	13		
No. Determination n	4		
Average Y=S_1/P (g/kg)	938.42		
S_r^2=S_3/P	19.7380	**Standard Deviation of Repeatability S_r**	4.4427
S_L^2=[(P*S_2-S_1^2)/P(P-1)]- S_r^2/n	107.8257	**S_L**	10.3839
S_R^2=S_r^2+S_L^2	127.5637	**Standard Deviation Reproducibility S_R**	11.2944
Repeatibility r=2.8*Sr	12.4397		
Reproducibility R=2.8*S_R	31.6243		
Relative Standard Deviation of Repeatability RSD_r=S_r*100/Y	0.4734		
Relative Standard Deviation of Reproducibility RSD_R=S_R*100/Y	1.2036		
Horwitz RSD_R (Hor)=2^[1-0.5*log(Y/1000)]	2.0192		
HorRat	0.596050802		

Table 8. Statistics of the results of TC2

S_1=sum Yi	12156.47		
S_2=sum Yi^2	11368278.71846		
S_3=sum Si^2	1009.0491		
No. Lab P	13		
No. Determination n	4		
Average Y=S_1/P (g/kg)	935.11		
S_r^2=S_3/P	77.6192	**Standard Deviation of Repeatability S_r**	8.8102
S_L^2=[(P*S_2-S_1^2)/P(P-1)]- S_r^2/n	31.3148	**S_L**	5.5960
S_R^2=S_r^2+S_L^2	108.9339	**Standard Deviation Reproducibility S_R**	10.4371
Repeatibility r=2.8*Sr	24.6685		
Reproducibility R=2.8*S_R	29.2240		
Relative Standard Deviation of Repeatability RSD_r=S_r*100/Y	0.9422		
Relative Standard Deviation of Reproducibility RSD_R=S_R*100/Y	1.1161		

Horwitz RSD_R (Hor)=2^[1-0.5*log(Y/1000)]	2.0203
HorRat	0.552461671

Table 9. Statistics of the results of TK1

S_1=sum Yi	9878.36		
S_2=sum Yi^2	7507393.27512		
S_3=sum Si^2	216.8846		
No. Lab P	13		
No. Determination n	4		
Average Y=S_1/P (g/kg)	759.87		
$S_r^2=S_3/P$	16.6834	**Standard Deviation of Repeatability S_r**	4.0845
$S_L^2=[(P*S_2-S_1^2)/P(P-1)]- S_r^2/n$	85.8376	**S_L**	9.2649
$S_R^2=S_r^2+S_L^2$	102.5210	**Standard Deviation Reproducibility S_R**	10.1253
Repeatibility r=2.8*Sr	11.4367		
Reproducibility R=2.8*S_R	28.3507		
Relative Standard Deviation of Repeatability $RSD_r=S_r*100/Y$	0.5375		
Relative Standard Deviation of Reproducibility $RSD_R=S_R*100/Y$	1.3325		
Horwitz RSD_R (Hor)=2^[1-0.5*log(Y/1000)]	2.0844		
HorRat	0.639270404		

Table 10. Statistics of the results of TK2

S_1=sum Yi	9910.97		
S_2=sum Yi^2	7557002.03672		
S_3=sum Si^2	314.5594		
No. Lab P	13		
No. Determination n	4		
Average Y=S_1/P (g/kg)	762.38		
$S_r^2=S_3/P$	24.1969	**Standard Deviation of Repeatability S_r**	4.9190
$S_L^2=[(P*S_2-S_1^2)/P(P-1)]- S_r^2/n$	81.4761	**S_L**	9.0264
$S_R^2=S_r^2+S_L^2$	105.6730	**Standard Deviation Reproducibility S_R**	10.2797
Repeatibility r=2.8*Sr	13.7733		
Reproducibility R=2.8*S_R	28.7833		

Relative Standard Deviation of Repeatability $RSD_r=S_r*100/Y$	0.6452
Relative Standard Deviation of Reproducibility $RSD_R=S_R*100/Y$	1.3484
Horwitz RSD_R (Hor)=2^[1-0.5*log(Y/1000)]	2.0834
HorRat	0.647208593

Table 11. Statistics of the results of SL1

S_1=sum Yi	5310.17		
S_2=sum Yi^2	2169581.13625		
S_3=sum Si^2	160.0977		
No. Lab P	13		
No. Determination n	4		
Average $Y=S_1/P$ (g/kg)	408.47		
$S_r^2=S_3/P$	12.3152	**Standard Deviation of Repeatability S_r**	3.5093
$S_L^2=[(P*S_2-S_1^2)/P(P-1)]- S_r^2/n$	39.8286	**S_L**	6.3110
$S_R^2=S_r^2+S_L^2$	52.1438	**Standard Deviation Reproducibility S_R**	7.2211
Repeatibility r=2.8*Sr	9.8260		
Reproducibility $R=2.8*S_R$	20.2190		
Relative Standard Deviation of Repeatability $RSD_r=S_r*100/Y$	0.8591		
Relative Standard Deviation of Reproducibility $RSD_R=S_R*100/Y$	1.7678		
Horwitz RSD_R (Hor)=2^[1-0.5*log(Y/1000)]	2.2885		
HorRat	0.77246925		

Table 12. Statistics of the results of SL2

S_1=sum Yi	5291.65		
S_2=sum Yi^2	2154402.28042		
S_3=sum Si^2	103.1793		
No. Lab P	13		
No. Determination n	4		
Average $Y=S_1/P$ (g/kg)	407.05		
$S_r^2=S_3/P$	7.9369	**Standard Deviation of Repeatability S_r**	2.8172
$S_L^2=[(P*S_2-S_1^2)/P(P-1)]- S_r^2/n$	34.0478	**S_L**	5.8350

$S_R^2=S_r^2+S_L^2$	41.9846	**Standard Deviation Reproducibility S_R**	6.4796
Repeatibility r=2.8*Sr	7.8883		
Reproducibility R=2.8*S_R	18.1428		
Relative Standard Deviation of Repeatability $RSD_r=S_r*100/Y$	0.6921		
Relative Standard Deviation of Reproducibility $RSD_R=S_R*100/Y$	1.5918		
Horwitz RSD_R (Hor)=2^[1-0.5*log(Y/1000)]	2.2897		
HorRat	0.695205688		

Using Grubb's test, a result for TC-1 from Lab13 was straggler, and a result for TC-2 from Lab13 was outlier. The data for TC-1 and TC-2 from Lab13 were eliminated, and a Grubb's test was run again. No straggler or outlier was found after the elimination. The analytical data for TC-1 and TC-2 after elimination is provided in Table 13.

Table 13 Statistics of the results of TC1 and TC2 after elimination of Grubb's test outlier and straggler

	TC1	TC2
Average Y	936.03	936.20
Number of Laboratories P	12	12
S_r	3.7428	4.6385
S_L	6.2095	5.7500
S_R	7.2503	7.3877
r	10.4799	12.9877
R	20.3009	20.6855
RSD_r	0.3999	0.4955
RSD_R	0.7746	0.7891
RSD_R(Hor)	2.0200	2.0199
HorRat	0.383457346	0.390658889

6. Conclusion

Fourteen different laboratories participated in this collaborative study. The analytical procedures from 1 laboratory significantly deviated from the proposed method, and multiple outliers were found in its results. Thus the results were excluded from statistical analysis. The results of the other laboratories are provided in Tables 1-6, and the statistical summary is included in Tables 7-12. The results for the samples evaluated are illustrated in Figures 1-6.

After Grubb's test, data for TC1 and TC2 from Lab 13 were found to contain outliers, so the data were re-analyzed after the outliers were omitted. These results were provided in Table 13.

Without elimination of any outliers or stragglers, the between lab experimental Relative Reproducibility Standard Deviation (% RSD_R) values of all six samples are below the calculated acceptable values based on the Horwitz curve calculation (% RSD_R (Hor)). With elimination of the outliers and stragglers, the % RSD_R is below % RSD_R (Hor) in all samples. The minimum number of considered results after elimination of stragglers and outliers was twelve. Horwitz ratio values obtained for this ethephon method collaborative trial are between 0.3 and 1.0, and considered acceptable.

Therefore, we consider this ethephon method as presented to be suitable. We recommend accepting this method as a provisional CIPAC method for the determination of ethephon in technical (TC and TK) and its associated formulated products (SL).

7. Chromatograms

Figure 7. HPIC chromatogram of blank

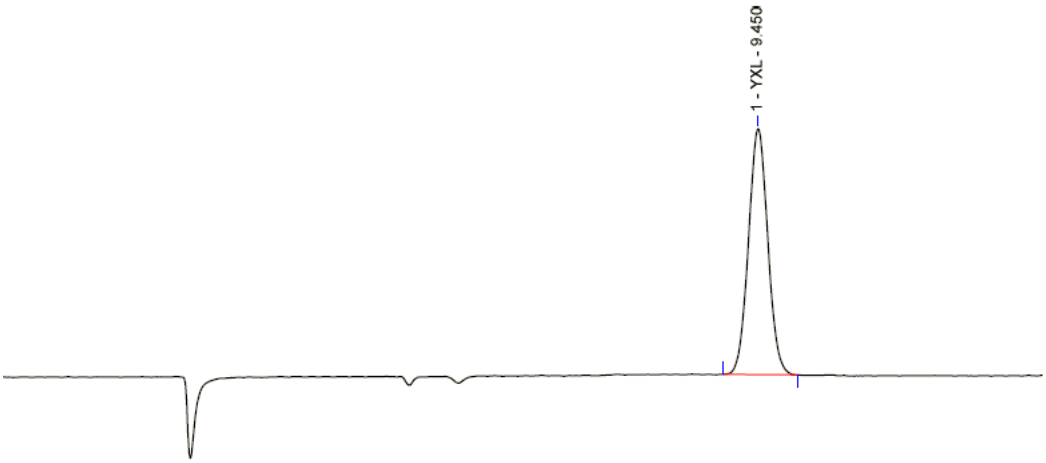

Figure 8. HPIC chromatogram of Ethephon standard

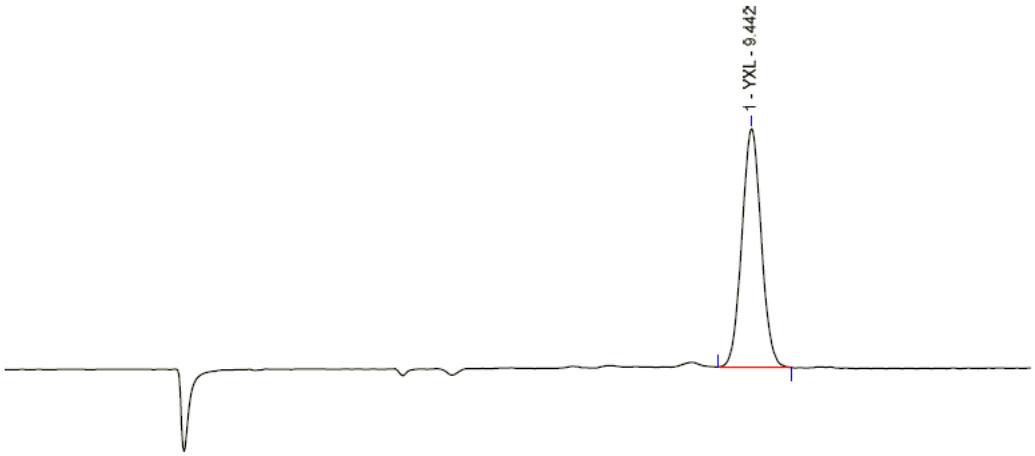

Figure 9. HPIC chromatogram of Ethephon TC

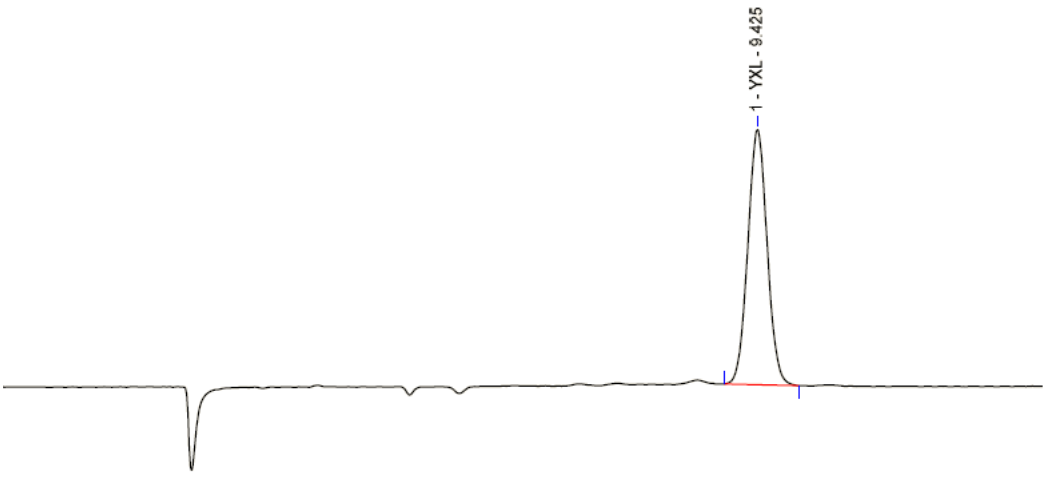

Figure 10. HPIC chromatogram of Ethephon TK

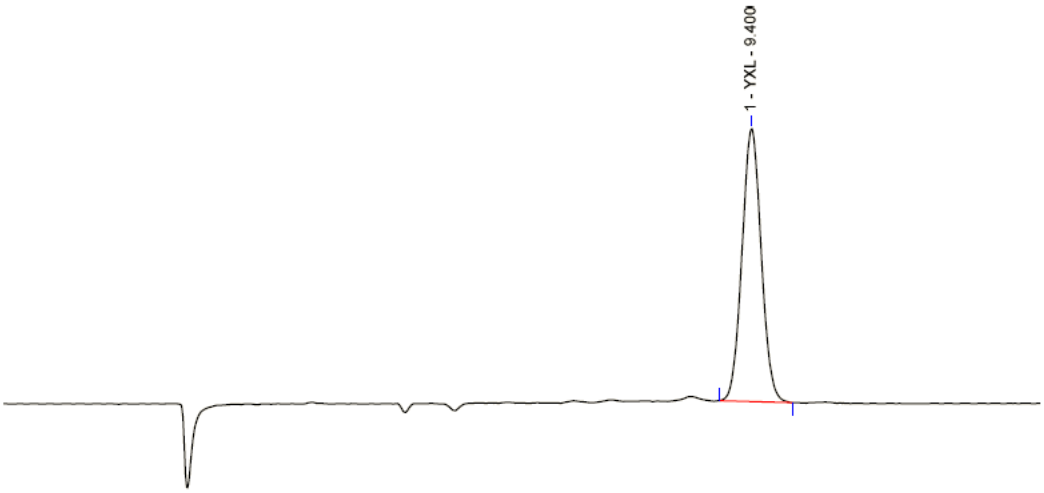

Figure 11. HPIC chromatogram of Ethephon SL